

Energy Recovery from Bio-Fuel Production through Two-Stage Anaerobic Co-Digestion Process

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Abstract. Anaerobic digestion of biodegradable matrices is regarded as a confirmed technology for energy recovery through biogas and its use in heat and power plants. Based on that, bio-hydrogen production in a two-stage digestion process can be considered as an optimization of the process to improve its efficiency. In this study, a two-stage digestion system was assessed and compared to the conventional one-stage process in terms of energy savings. Primary Energy Saving was evaluated in relation to different biogas users, using as input data gas production coming from a pilot scale semi-continuous test. Splitting the process into two different phases was demonstrated to be functional for improving anaerobic performances with an enhancement in biogas production and methane content in the methanogenic reactor. Considering different feedstocks, the two-stage co-digestion process of a mixture of organic waste and wastewater sludge proves to be the most virtuous with regards to energy savings. In the two-stage co-digestion system with ICE as biogas user, energy savings increased by 57.7% compared to the one-stage configuration.

1. INTRODUCTION

The anaerobic digestion (AD) process converts biodegradable substrates into a renewable energy source, biogas, and into a residual matter used as a fertilizer and soil conditioner, namely digestate. For these reasons, AD represents a proven technology for bio-fuel and bio-product production by means residues valorization [1]. Turning bio-waste into new resources is one of the aims of the EU action plan for the Circular Economy for the development of a sustainable and low-carbon future [2].

Most of the AD capacity lies in wastewater treatment plants (WWTPs), where AD is employed for stabilizing wastewater sludge. Considering the low organic loading rate and biogas yields of sludge, energy recovery via AD is usually not sufficient to cover WWTPs. A feasible solution to improve digestion efficiency and optimize AD process conditions is the co-digestion of different biodegradable substrates, using the spare digestion capacity in WWTPs [3], [4], [5], [6], [7]. An optimal co-substrate for wastewater sludge (WWS) could be considered the sorted organic fraction of municipal solid waste (OFMSW), due to its wide availability in municipal areas and its easily biodegradable nature [8], [9], [10]. Therefore, the possibility of recovering organic waste both for the production of bio-based products and for the production of a clean renewable fuel, increases the interest in using it as a co-substrate for the digestion process [11]. In a co-digestion feedstock obtained through a mixture of OFMSW and WWS, both substrates provide important contributions to the AD process and lead to the improvement of process efficiency. The OFMSW influences process kinetics and supplies carbon source, while WWS increases the system's buffer capacity [7], [12], [13].

In addition to this, the two-stage AD digestion process could be considered another promising technology to improve process efficiency. This method increases energy conversion efficiency by producing two different high-

energy gas streams by splitting the conventional AD process into two reactors in series with one another, the fermentative reactor and the methanogenic one [14]. In the fermentative reactor, called Dark Fermentation (DF) reactor, the acidogenic phase of AD is performed and a hydrogen-rich biogas is produced together with a consistent release of Volatile Fatty Acids (VFAs) in the digestate. In the methanogenic reactor, the VFAs and the remaining organic matter are gradually converted into a gas stream with a high concentration of methane [15]. The two-stage AD process has been shown to enhance gasification efficiencies in the second reactor due to preliminary degradation of organic substrates in the fermentative reactor, making it more easily accessible to the methanogenic bacteria than the one-stage AD process [16], [17], [18], [19]. On this basis, DF could be seen as a biological pre-treatment step for the subsequent AD process [14], [20], [21], [22], [23].

This study is a development of the research of Baldi et al. [12], where one-stage and two-stage AD processes were performed for different feedstocks with the aim of evaluating the performance of the process in terms of bio-fuel production. Previous studies [1], [20], [24] have compared similar scenarios for the assessment of energy recovery of gas flows obtained through the one-stage and two-stage AD processes. The objective of this work is to compare the energy performances related to Primary Energy Saving of different biogas users for different AD scenarios. Experimental data related to energy production of different feedstock, OFMSW as sole substrate and a mixture of OFMSW and WWS as a co-digestion substrate, are considered in this study. Four different scenarios with several biogas users are evaluated for both the digestion process (with OFMSW as substrate) and the co-digestion process (with OFMSW+WWS as substrate). In two scenarios the traditional one-stage AD process was considered, while in the other two scenarios, the two-stage process was performed by adding a fermentative digester as a preliminary step.

Data were collected from different sources: methane and hydrogen production data were obtained by carrying out semi-continuous tests in one and two-stage systems [12], while other data were provided directly from the WWTP management data considered as a case study [1], [24].

2. MATERIALS AND METHODS

2.1. Feedstock and Inoculum Characterization

OFMSW and WWS were used as raw materials separately or mixed together respectively to simulate the digestion and co-digestion process. OFMSW was sampled from approximately 10 tons of source-separated organic waste coming from a kerbside collection system of a Tuscan (Italy) municipality. The sample was immediately treated with a food processor (Problend 6, Philips, Netherlands) and mixed with tap water to reach a total solid content of about 20 % w/dw. The final OFMSW slurry was then stored in plastic containers at -20°C until use. Concerning WWS, 200 liters of activated sludge was taken from the aerobic unit of the WWTP considered as reference plant in this study. The sample of WWS was stocked in plastic tanks and put in the refrigerator at 4°C.

As an inoculum for the semi-continuous trials, the same WWS sample was used for the DF reactor. In order to inhibit hydrogenotrophic methanogen bacteria in the DF reactor and select only the hydrogen producing bacteria, the fermentative inoculum was heat-treated at 80°C for 30 minutes before starting the experimental tests [25], [26]. Inoculum treatment was carried out in 250 ml beakers set into a static oven (UM200, Memmert GmbH, Germany) and continuously measuring the temperature of the inoculum by means of a digital thermometer (T1, Testo S.p.A., Italy). With regard to the methanogenic reactor, a digested sludge provided from a wet mesophilic anaerobic digester treating organic waste and cattle manure (IN_AD) was instead used as inoculum.

Before storage, each sample was characterized in terms of Total Solids (TS), Total Volatile Solids (TVS) and pH following standard procedures [27]. OFMSW, WWS and IN_AD characteristics are shown in Tab. 1.

TABLE 1. Substrates and inoculum characterization. Values are expressed as average and standard deviations (n=3).

| | TS (%) | TVS/TS (%) | pH |
|--------------|------------|------------|-----------|
| OFMSW Slurry | 19.9 ± 0.6 | 80.6 ± 0.9 | 3.8 ± 0.1 |
| WWS | 2.1 ± 0.0 | 79.3 ± 0.3 | 7.1 ± 0.0 |
| IN_AD | 2.6 ± 0.0 | 61.9 ± 0.4 | 8.2 ± 0.1 |

With the aim of depicting a wet digestion technology and obtaining a feedstock mesh suitable for this technology, the OFMSW was diluted in order to achieve a total slurry solid content of approximately 5% by weight. Therefore, to perform the experiments under wet digestion conditions, OFMSW slurry was daily mixed in the food processor with water or WWS respectively for digestion and co-digestion experiments. In both cases, the ratio of OFMSW slurry with water or WWS was approximately equal to 1:5 by weight.

2.2. Experimental Set-up: Semi-Continuous Trials

The experimental data used in this study were obtained from one-stage and two-stage AD process tests performed by Baldi et al. [12] on a pilot scale. In particular, bio-hydrogen and bio-methane production was evaluated employing two Continuous Stirred Tank Reactors (CSTR) used for the fermentative and methanogenic phase (Fig. 1). The fermentative stage (DF) was carried out using a stainless steel (AISI 316) reactor with a volume of 6 liters (3 liters working volume). The methanogenic stage (AD) was performed in a similar reactor with a volume of 20 liters (working volume 12 liters). Inside the reactors the medium was continuously mixed by a mixing blade connected to an electric motor (COAX MR 615 30Q 1/256, Unitec S.r.l., Italy). The mesophilic conditions were ensured by a water jacket where warm water passed through. A water bath warmed up by a thermostat (FA90, Falc Instruments S.r.l., Italy) guaranteed a constant water temperature of 37° C. pH probes (InPro4260i, Mettler Toledo, Italy) measured pH continuously in each reactor. In the fermentative reactor, a dedicated pH control system gave assurance of pH control by dosing of NaOH 2M solution using a peristaltic pump (Reglo ICC, Ismatec, Germany). Volumetric counters connected to the top of each reactor measured the volume of the biogas produced, which filled the 10-liter multi-layer foil bags (SuperITM, Merck KGaA, Germany) attached to them. The analysis of the biogas composition was then performed by gas-chromatography analysis (3000 Micro GC, INFICON, Switzerland) in terms of H₂, CH₄, CO₂, N₂, O₂ and H₂S contents.

Feedstock mashes were daily filled into the reactors through a syringe and subsequently the same amount of digestate was collected. The experimental trials were performed in order to reproduce both the one-stage and two-stage AD process. At the beginning, the AD reactor run alone for the evaluation of the conventional one-stage process. In a second step, the two reactors were connected in series with each other and the digestate coming from the preliminary DF reactor was transformed into the substrate for the subsequent AD reactor. This configuration aimed at assessing the performance of the two-stage AD process. The operating conditions applied during the experimental test were the same as those considered in the study of Baldi et al. [12]. Process stability was daily monitored by VFAs measurement on the outgoing digestate using a gas chromatograph and anaerobic performance was assessed in terms of Specific Gas Production (SGP) and methane and hydrogen content in biogas.

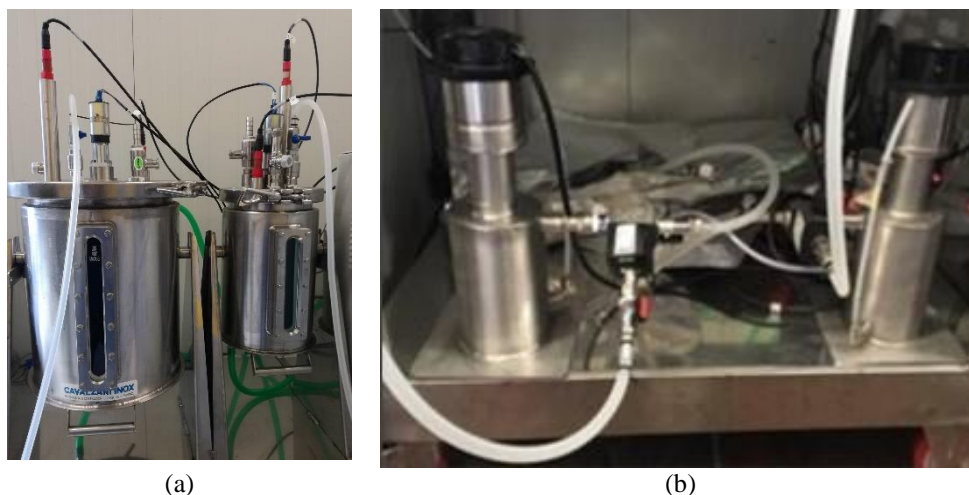


FIGURE 1. CSTR pilot scale reactors used for the experimental tests (a) and volumetric counters (b).

2.3. Inventory Analysis: Fermentation and Co-Digestion Scenarios

The present study regards the evaluation of the Primary Energy Savings of the digestion and co-digestion process using as feedstock respectively the sole OFMSW and a mixture of OFMSW+WWS. One-stage and two-stage AD process were carried out for each substrate in order to compare the production performance of the bio-fuels. For each substrate, four different scenarios were correlated with each other in terms of energy saving performance considering the utilization of the bio-fuels through different biogas users. In two scenarios, Scenario AD_ICE and Scenario AD_GT, the conventional one-stage AD process was taken into account referring to methane production only. Two-stage AD process was evaluated in two other scenarios, Scenario DF+DA_ICE and Scenario AD+AD_GT, considering the production of hydrogen with a fermentative DF reactor in addition to AD reactor.

Several biogas users were utilized for energy recovery analysis concerning both the electric and thermal energy. In Scenario AD_ICE and Scenario DF+AD_ICE, Internal Combustion Engine (ICE) was assessed to recover the biogas flows extracted from each reactor and to cogenerate electric and thermal energy. Considering the hydrogen-rich gas flow obtained from the latter case, the ICE was combined with a Molten Carbonate Fuel Cell (MCFC) to produce electricity. The AD_GT and DF+AD_GT scenarios evaluate energy savings by taking into account a Gas Turbine (GT) as biogas user. In Fig. 2 the performed digestion and co-digestion scenarios are shown.

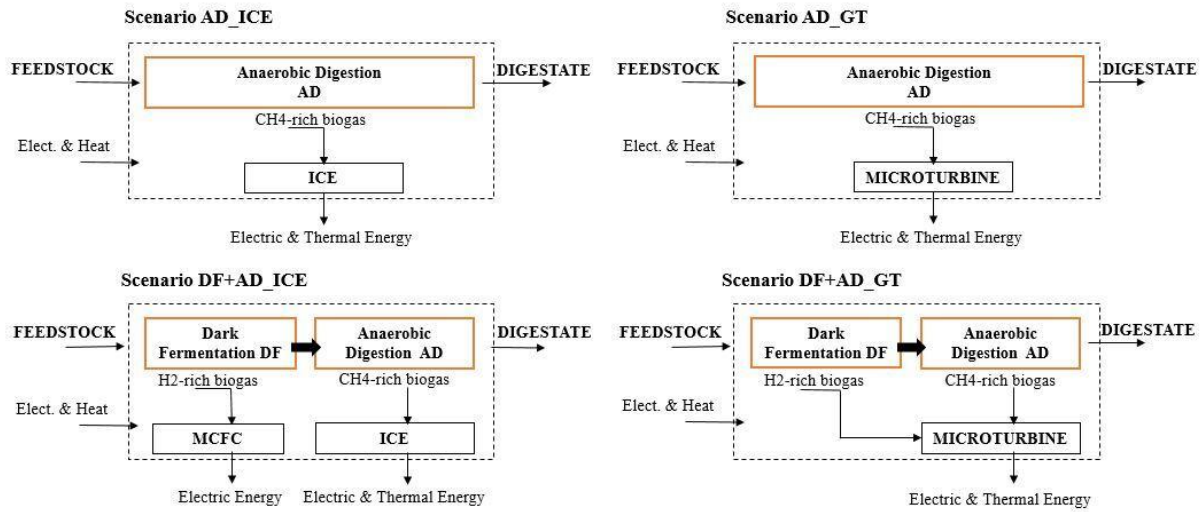


FIGURE 2. Scenarios layout for digestion and co-digestion process.

In order to estimate the benefit of energy savings for each scenario, the Primary Energy Saving (PES) was calculated referring to Eq. 1, in according to Directive 2012/27/UE [28]

$$\text{Primary Energy Saving} = \frac{E_{el}}{\eta_{el,rif} * \rho_g} + \frac{Q_{th}}{\eta_{th,rif}} \quad (1)$$

where E_{el} and Q_{th} represent the net electric and thermal energy produced and recovered in each scenario respectively, $\eta_{el,rif}$ is the reference electricity efficiency coefficient considered equal to 0.525, ρ_g is the distribution losses coefficient for a value of 0.963 and $\eta_{th,rif}$ constitutes the thermal energy reference efficiency coefficient equal to 0.900. With regard to the inventory data for the evaluation of energy saving, for each digestion and co-digestion scenario, the mass and energy balances were estimated. Inventory data related to electric consumptions were provided by the operational data of the WWTP considered as a case study. The consumptions due to the pre-treatment section of OFMSW performed by means of a screw-press and a cleaning system were also taken into account. Thermal energy utilization was evaluated with reference to heat needs and losses for warming the digesters, that work at mesophilic conditions. In Tab. 2 the data assumed for each scenario to assess the mass and energy balances are reported.

TABLE 2. Inventory data for digestion and co-digestion process.

| Digestion (OFMSW) | | | | |
|----------------------------------|---|---|--|---|
| | Scenario AD_ICE | Scenario AD_GT | Scenario DF+AD_ICE | Scenario DF+AD_GT |
| Treated flow (t/d) | 234 | 234 | 265 | 265 |
| Rector volume (m ³) | 4,617 | 4,617 | 993 (DF) 4,617 (AD) | 993 (DF) 4,617 (AD) |
| HRT (d) | 17 | 17 | 3 (DF) 12.8 (AD) | 3 (DF) 12.8 (AD) |
| OLR (kgTVS/m ³ *d) | 2.1 | 2.1 | 14.2 (DF) 2.1 (AD) | 14.2 (DF) 2.1 (AD) |
| Co-Digestion (OFMSW+ WWS) | | | | |
| | Scenario AD_ICE | Scenario AD_GT | Scenario DF+AD_ICE | Scenario DF+AD_GT |
| Treated flow (t/d) | 246 | 246 | 339 | 339 |
| Rector volume (m ³) | 4,617 | 4,617 | 1,272 (DF) 4,617 (AD) | 1,272 (DF) 4,617 (AD) |
| HRT (d) | 17 | 17 | 3 (DF) 12.8 (AD) | 3 (DF) 12.8 (AD) |
| OLR (kgTVS/m ³ *d) | 2.4 | 2.4 | 14.0 (DF) 2.4 (AD) | 14.0 (DF) 2.4 (AD) |
| Biogas utilization | | | | |
| | Scenario AD_ICE | Scenario AD_GT | Scenario DF+AD_ICE | Scenario DF+AD_GT |
| Bio-fuel users | ICE $\mu_{el} = 0.42$ $\mu_{th} = 0.43$ | Gas Turbine $\mu_{el} = 0.33$ $\mu_{th} = 0.39$ | MCFC (DF) $\mu_{el} = 0.45$ ICE (AD) $\mu_{el} = 0.42$ $\mu_{th} = 0.43$ | Gas Turbine $\mu_{el} = 0.33$ $\mu_{th} = 0.39$ |
| Functioning hours (h/y) | 7,992 | 7,992 | 7,992 (MCFC) 7,992 (ICE) | 7,992 |

3. RESULTS AND DISCUSSION

3.1. Digestion and Co-Digestion Performance

Based on the above background, the two-stage digestion process was assessed and compared with the conventional one-stage anaerobic system. Two different substrates were used to simulate the digestion and co-digestion process in order to assess the performances of the process related to bio-H₂ and bio-CH₄ production. The experimental data considered for comparison were referred to the steady state, reached after the acclimatization phase.

Tab. 3 shows the CSTR results achieved in the study of Baldi et al. [12], taken as input data for the energy saving evaluation assessed in this study. The anaerobic performances are expressed in terms of biogas production (SGP) and quality (H₂ and CH₄ content).

TABLE 3. CSTR test results for digestion and co-digestion experimental tests. Values are expressed as average and standard deviations.

| Digestion (OFMSW) | | | |
|--------------------------|--------------|-------------|--------------|
| | One-stage | Two-stage | |
| | AD reactor | DF reactor | AD reactor |
| SGP (NL/kgTVS*d) | 694.4 ± 24.6 | 43.1 ± 12.8 | 704.6 ± 28.5 |
| H ₂ (%) | - | 22.9 ± 5.5 | - |
| CH ₄ (%) | 65.2 ± 1.9 | - | 68.4 ± 1.1 |
| Co-Digestion (OFMSW+WWS) | | | |
| | One-stage | Two-stage | |
| | AD reactor | DF reactor | AD reactor |
| SGP (NL/kgTVS*d) | 485.9 ± 25.8 | 44.8 ± 12.6 | 611.0 ± 45.4 |
| H ₂ (%) | - | 18.4 ± 6.3 | - |
| CH ₄ (%) | 61.2 ± 2.2 | - | 70.1 ± 1.6 |

From the above results, an increment in the methane content of the biogas collected by AD reactor in the two-stage process of 3.2% and 8.9% respectively for the digestion and co-digestion process can be noted. Compared to the conventional one-stage process, the two-stage system enabled an enhancement in methane production in the second reactor, which also corresponds to an increase in biogas generation. This improvement in biogas production and quality was due to the better hydrolysis of the feedstock that took place in the fermentative reactor. As shown in Fig. 3, the VFAs were produced almost exclusively in the DF reactor due to the improvement of substrates hydrolysis in that stage. The subsequent methanogenic stage in the two-stage process allowed them to be almost completely degraded. The relevant amount of VFAs production in the first reactor, supplied readily available organic acids into the methanogenic reactor, which caused an increase in biogas production and methane content [16], [17], [18], [19]. Moreover, the biogas production increment is also due to the presence of the first fermentative reactor where a hydrogen-rich biogas is produced and contributed to the increased biogas generation. Based on that, the improvement of the biogas quality through a two-stage process represents a possibility for the optimization of biogas upgrading systems. Recently, several experimental and industrial scale upgrading systems were considered to assess the feasibility of converting biogas into bio-methane [29], [30], [31].

Considering the total biogas production in the two-stage process, determined by the sum of the biogas coming from the first and second digester, the percentage of biogas rose to 7.7% and 35.0% respectively for digestion and co-digestion. The difference in percentage increase is mainly due to the characteristics of feedstock. OFMSW slurry represents a substrate with a high carbohydrate content while co-digestion feedstock, consisting of a mixture of OFMSW+WWS, presents a lower content of carbohydrates due to the presence of WWS [25], [32], [33], [34]. Feedstock composition influences the performance of AD process with respect to SGP and methane content, especially due to the different biodegradability rate of the matrices.

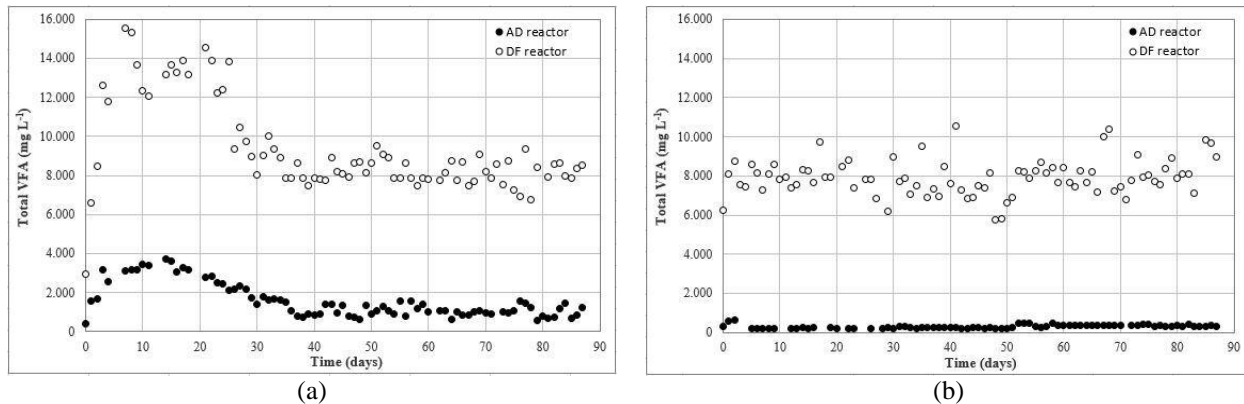


FIGURE 3. Total VFAs content in DF and AD reactor during the digestion (a) and co-digestion test (b).

3.2. Primary Energy Saving Performances

Table 4 reports the results of the mass and energy balance for all scenarios of the digestion and co-digestion process. In the following Fig. 4, the different layouts are compared to each other in terms of energy saving.

TABLE 4. Mass and energy balance for digestion and co-digestion process.

| Digestion (OFMSW) | | | | |
|--------------------------------------|----------------------------|---------------------------|--------------------------------|--------------------------------|
| | Scenario AD_ICE | Scenario AD_GT | Scenario DF+AD_ICE | Scenario DF+AD_GT |
| Bio-fuels production | | | | |
| Biogas (Nm ³ /y) | 2,155,119 | 2,155,119 | 177,381 (DF) 2,185,081 (AD) | 177,381 (DF) 2,185,081 (AD) |
| H ₂ (Nm ³ /y) | - | - | 40,620 | 40,620 |
| CH ₄ (Nm ³ /y) | 1,405,138 | 1,405,138 | 1,494,595 | 1,494,595 |
| EE - Electric Energy (MWh/y) | | | | |
| Input EE | 2,344 | 2,304 | 2,511 | 2,458 |
| Output EE | 5,730 | 4,523 | 6,153 | 4,929 |
| Net EE | 3,385 | 2,219 | 3,643 | 2,471 |
| TE – Thermal Energy (MWh/y) | | | | |
| Input TE | 2,074 | 2,074 | 3,089 | 3,089 |
| Output TE | 5,894 | 6,108 | 6,269 | 6,108 |
| Net TE | 3,385 | 2,219 | 3,643 | 2,471 |
| Co-Digestion (OFMSW+WWS) | | | | |
| | Scenario AD_ICE | Scenario AD_GT | Scenario DF+AD_ICE | Scenario DF+AD_GT |
| Bio-fuels production | | | | |
| Biogas (Nm ³ /y) | 1,748,538 | 1,748,538 | 232,991 (DF) 2,194,075 (AD) | 232,991 (DF) 2,194,075 (AD) |
| H ₂ (Nm ³ /y) | - | - | 42,870 | 42,870 |
| CH ₄ (Nm ³ /y) | 1,070,105 | 1,070,105 | 1,538,046 | 1,538,046 |
| EE - Electric Energy (MWh/y) | | | | |
| Input EE | 2,303 | 2,304 | 2,516 | 2,458 |
| Output EE | 4,363 | 3,445 | 6,334 | 5,076 |
| Net EE | 2,060 | 1,141 | 5,076 | 2,618 |
| TE – Thermal Energy (MWh/y) | | | | |
| Input TE | 2,156 | 2,156 | 3,814 | 3,814 |
| Output TE | 4,489 | 6,108 | 6,452 | 6,108 |
| Net TE | 2,333 | 3,952 | 2,638 | 2,294 |

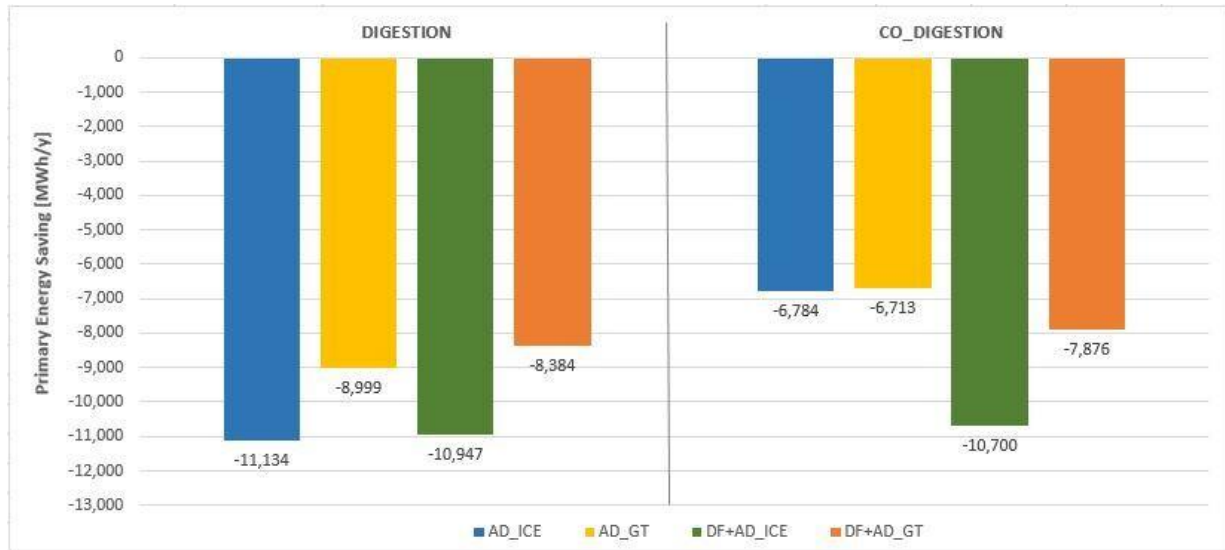


FIGURE 4. Digestion and co-digestion performance comparison.

The results illustrated that splitting the AD process into two reactors gives beneficial effects on primary energy saving compared to the traditional AD digestion process. This result is more evident in the co-digestion process where the presence of the DF stage improves biogas production by 35% in the second reactor. For each scenario it can be noted that the production of electric energy benefits from the fermentation phase. Concerning thermal energy, the presence of the new DF digester increased thermal consumptions due to the demand of more thermal energy for heating the added digester. In this case, the enhancement of the biogas quality and methanogenic phase gas production influenced the energy balance. For the digestion process, the 7.7% increase in biogas is not sufficient to cover the additional thermal energy required, demonstrating that the two-stage process is not as efficient as the conventional one. In fact, the AD_ICE scenario for OFMSW slurry digestion results to be the best scenario relative to the energy saving values. On the contrary, in the co-digestion process the 35% increment in the bio-fuel production guaranteed energy balance and the DF+AD_ICE scenario with a two-stage process is the most efficient scenario. Energy recovery is influenced by the enhancement of biogas production due to the DF stage. Anaerobic performance, expressed in terms of primary energy savings, showed that biogas increment balances the extra heat energy required only in the co-digestion process, which has the better energy performance.

4. CONCLUSIONS

The two-stage co-digestion process of OFMSW and WWS presents the best performance in terms of energy recovery. The introduction of a preliminary fermentative reactor efficiently improved anaerobic performance in the methanogenic phase compared to the conventional one-stage reactor system. The increment in biogas production and quality is due to the optimization of the metabolism of hydrolytic bacteria in the fermentative reactor that determines an increase in the production of VFAs readily available in the methanogenic reactor. The performances of the co-digestion process were higher than that of the two-stage digestion of OFMSW as sole substrate. The overall SGP in the two-stage process increased by 35% and 7.7% respectively for the co-digestion and digestion process, with a corresponding increment in methane content of 8.9% and 3.2%.

Considering the bio-fuel production obtained by CSTR tests, it was possible to estimate the performances of the process in terms of energy saving by comparing different biogas users. The energy advantages of the additional reactor results in the balancing out of the additional thermal energy consumptions due to the new reactor only for the co-digestion configuration. The assessment reports that the two-stage co-digestion process with ICE and MCFC as biogas users is the most virtuous with regards to energy savings. In conclusion, dark fermentation can be considered as a suitable pre-treatment to improve the anaerobic co-digestion of OFMSW and WWS and provide primary energy savings. In the two-stage co-digestion system with ICE as biogas user, energy savings increased by 57.7% compared to the one-stage configuration.

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